

## Appendix A: Quality Assurance

### Background

The National Coastal Assessment (NCA) program monitors and assesses the quality of the data that is collected through the activities of the NCA Quality Assurance (QA) program. The NCA QA program is conducted under the guidance of the National Health and Environmental Effects Research Laboratory (NHEERL) Director of Quality Assurance. The NCA QA team consists of:

- National Quality Assurance Coordinator – Assures a quality assurance program is in place and being followed as well as documentation of the known quality of the data sets developed by the National Contract Laboratories;
- Four regional quality assurance coordinators – Assure that the quality assurance program is being followed and develop the documentation supporting the known quality of the data collected in NCA; and,
- Twenty-four state quality assurance coordinators – Responsible for reviewing and qualifying all data sets sent to the program from their respective states.

A detailed Quality Assurance Project Plan was developed by NCA (U.S. EPA 2001b) and provided to all participants in the program. Compliance with the QAPP is assessed through extensive field training exercises, site visits, reviews, and audits. The QAPP addresses multiple levels of the program. These range from the collection of field samples and laboratory processing of these samples, to the review of data sets compiled from the field and laboratory activities. The NCA QA team is responsible for performing assessments of the adequacy of these activities.

### 1999/2000 Survey

The NCA enacted a diverse panel of environmental scientists to help formulate a list of core indicators to help ensure that the NCA collected the appropriate types of data to support its mission. In order to ensure that the data collected were of appropriate quality to generate sound estimates on environmental condition, the NCA utilized the U.S. EPA's concept of Data Quality Objectives (DQOs) to set the overall level of data quality required by management to make informed decisions. In other words, how much error can be tolerated within the measurement process before the data are deemed unacceptable?

The NCA program developed an *a priori*, program-level DQO for status estimates: **“For each indicator of condition, estimate the portion of the resource in degraded condition within  $\pm 10\%$  for the overall system and  $\pm 10\%$  for subregions with 90% confidence based**

**on a completed sampling regime.”** This requirement was met by all of the indicators used for the 1999 to 2000 estimates with the exception of Puerto Rico. The NCA design never intended to treat Puerto Rico’s samples as a sole measure of the condition of the Caribbean and Pacific island commonwealths. Once other commonwealth islands are included in the NCA surveys, the uncertainty associated with condition estimates will be reduced significantly. The level of uncertainty (error) associated with the individual indicators for each region and the national estimates (Table A-1) ranges from 1% to 16% (including Puerto Rico) and 1-9% (excluding Puerto Rico). The uncertainty associated with areal estimates of ecological condition in the Great Lakes cannot be determined.

**Table A-1.** Levels of Uncertainty Associated with the Estimate of Proportional Area Exceeding the Indicator Criteria

Indicator	NE	SE	Gulf	West	Great Lakes	Puerto Rico	United States
Water Quality Index	5%	4%	8%	4%	NA	15%	5%
Water Clarity	5%	5%	9%	3%	NA	15%	4%
Nitrogen	5%	<1%	5%	3%	NA	14%	3%
Phosphorus	6%	5%	8%	3%	NA	8%	4%
Chlorophyll <i>a</i>	5%	4%	9%	4%	NA	14%	5%
Dissolved Oxygen	3%	4%	4%	4%	NA	8%	3%
Sediment Quality Index	5%	3%	4%	4%	NA	15%	4%
Sediment Contaminants	4%	1%	8%	5%	NA	10%	3%
Sediment Toxicity	4%	6%	7%	4%	NA	10%	2%
Sediment TOC	2%	6%	8%	4%	NA	16%	5%
Wetland Loss	<.1%	<.1%	<.1%	<.1%	NA	NA	<.1%
Benthic Index/Equivalent	5%	5%	9%	4%	NA	15%	5%
Fish Contaminant Index	6%	5%	5%	5%	NA	NA	4%
Aquatic Life Use Impairment	2%	3%	3%	3%	NA	8%	2%
Human Use Impairment	4%	5%	5%	5%	NA	NA	4%
Unimpaired	2%	3%	3%	4%	NA	9%	2%

Data from the NCA 1999/2000 survey were evaluated and appropriately qualified based on the projects Measurement Quality Objectives (MQO’s). MQO’s establish the quality goals for the individual measurements taken by the program used in the generation of condition indicators utilized by NCA. Approximately 90% of the data collected, processed, and generated for this report fully met the MQO requirements stated in the NCA-QAPP. Nine percent of the data partially met the requirements; therefore it was qualified and only conditionally used. Only 1% of the data failed to meet the requirements and were not used. The conditional use of the

data was only allowed after it was determined that the data would not significantly bias the results.

### *Field Collections*

NCA conducted a 4-5 day training workshop for all states participating in the program (Table A-2). The workshop included training on the application of the probability—based design to state monitoring activities and standardized methods required for sample collection. State field crews were evaluated on their ability to apply the protocols and received certification after the training based on a field trial. A sample matrix of the state training activities is shown in Table A-3.

Upon initiation of field activities, the field crews from Mississippi, Maine, Delaware, New York, New Jersey, and Rhode Island were audited and found to be compliant with the QAPP. The field crews from Washington and Oregon were observed by a representative from EPA Region 10 and the observations were documented. These observations were evaluated by the NCA QA Coordinator with no findings.

**Table A-2.** Number of Individuals Trained by NCA in 1999/2000 for Each of the Participating States

Region	State or Agency	Number Trained
West Coast	CA	4
	OR	5
	WA	5
	NOAA/NMFS	6
Gulf of Mexico	TX	13
	LA	10
	MS	2
	AL	5
	FL	7
Southeast	GA	2
Northeast	MA	10
	ME	10
	DE	10
	NH	10
	NY	10
	NJ	10
	CT	10
	RI	10

**Table A-3.** Matrix of training activities for the northeast region of NCA in 2000.

Subject	ME	NH	MA	RI	CT	NY	NJ	DE
Intro to C2000	yes	yes	yes	yes	yes	yes	yes	yes
List of indicators	yes	yes	yes	yes	yes	yes	yes	yes
Station and sample Ids, barcodes	yes	yes	yes	yes	yes	yes	yes	yes
Locating stations	yes	yes	yes	yes	yes	yes	yes	yes
Station datasheet	yes	yes	yes	yes	yes, but may not use it	yes	yes	yes
CTD profile	Include instrumentation	yes	General only	yes	General only	yes	General only	General only
PAR profile	Include instrumentation	yes	yes	yes	yes	yes	yes	yes
Secchi Depth	yes	yes	yes	yes	yes	yes	yes	yes
Nutrients	Full detail	Full detail	general only	Full detail	General only	Mixed	Full detail	Full detail
Benthic infauna	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail
Sediment Chemistry	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail
Sediment Toxicity	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail
Trawl Operations	Full detail	Full detail	Full detail	Full detail	General only	Full detail	General only	Full detail
Fish Community	Full detail	Full detail	Full detail	Full detail	General only	Full detail	General only	Full detail
Fish Pathology	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail
Fish Chemistry	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail
Shipping	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail
Computer System	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail	Full detail

## Laboratory Analyses

Prior to the analyses of any samples in 1999, the analytical laboratories from Washinton, Oregon, and California had to perform a demonstration of capability. Each laboratory was sent a set of Standard Reference Materials (SRMs) as unknown samples for analysis. These samples represented both organic and inorganic compounds in sediment and tissue matrices (Table A-4) which were representative of the type of samples NCA would be providing them. The results from these analyses were evaluated in order to determine whether the lab was capable of correctly identifying and quantifying the analytes of interest within the QA requirements outlined in the NCA QAPP. In lieu of analyzing the SRMs, each lab could submit its results from the National Institute of Standards and Technology (NIST) annual inter-laboratory comparison (ILC), a program examining performance-based quality assurance among multiple laboratories using NIST-generated sediment and tissue contaminant samples of known concentrations. These samples are analyzed by participating laboratories using a variety of methods and the results are compared to the known concentrations.

**Table A-4.** Standard Reference Materials sent to Washington, Oregon, and California state laboratories for a demonstration of capability.

SRM	Matrix	Class of Compounds
CRM 2976	Mussel Tissue	inorganics
CARP-1	Fish Tissue	organics
MESS-2	Marine Sediment	inorganics
SRM 1944	NY/NJ Waterway Sediment	organics

### California

Two separate laboratories performed the chemical analyses of samples for the state of California. The laboratory performing the organic analyses submitted the required SRMs for evaluation, while the laboratory performing the inorganic analyses submitted their NIST ILC results. The inorganics laboratory satisfactorily demonstrated technical capability for metals analyses by submitting their results for the current NOAA/NIST interlaboratory calibration exercise. In fact, the NOAA/NIST exercise included samples identical to those distributed by NCA. For both matrices, the laboratory generally exceeded NCA's quality criteria for accuracy,  $\pm 20\%$  agreement to the accepted true concentration (only applies to those analytes with accepted true values greater than 10 times laboratory's method detection limit [MDL]). The laboratory also demonstrated a high degree of precision for the three replicate analyses conducted with each sample. The organics laboratory satisfactorily demonstrated technical capability for pesticide, PAH and PCB analyses with the successful analysis of the CARP-1 and SRM-1944. The percent recoveries and reported MDL's for the required analytes met or exceeded the NCA quality criteria.

### Washington

The laboratory performing the analyses for the state of Washington submitted results from the analysis of the SRMs for inorganics and results from the NIST ILC for organics. The laboratory's results for Marine Sediment VIII (QA98SED8) were indicative of the laboratory capability to produce high quality analytical data for organic contaminants in sediments and met with NCA's general expectation for technical competency.

The laboratory's results for the inorganic SRMs, CRM-2976 and MESS-2, demonstrated that they had the capability to successfully analyze sediment and tissue samples for metals. Results and MDL'S provided were within the general criteria for technical competence required by NCA.

## Oregon

The laboratory performing the sample analyses for the state of Oregon submitted their results from analysis of the SRMs for evaluation of capability for both organic and inorganic analyses. The results submitted by the laboratory for the sediments appear marginal when gauged against NCA's established acceptability criteria. For analytes with true/accepted (SRM) concentrations greater than 10 times the laboratory's reported MDL, the laboratory's submitted values should be within  $\pm 35\%$  of the accepted value (including the confidence limits) for at least 70% of the analytes within a class of compound (e.g., PCBs). It is not uncommon for a laboratory to encounter difficulty in meeting these strict standards. Because continued improvement was anticipated, the laboratory was conditionally approved to initiate the analyses of sediment samples with the understanding that all results for field samples will be critically reviewed regarding NCA quality standards. If these standards were not met, the data were flagged or even dropped altogether from the Regional and National Databases.

The results from the analyses of the SRMs, CRM-2976 and MESS-2, generally met with the NCA quality standard for relative accuracy, agreement within  $\pm 20$  percent of the accepted true value for each analyte.

## Other Coastal States

In 2000, nineteen additional coastal states became partners in the National Coastal Assessment. Many of the states did not wish to, or were not capable of analyzing the samples that were being collected. In order to meet the need for a centralized laboratory processing facility, NCA established national contracts in which commercial laboratories were contracted to perform the required analyses. The management of the contracts, coordination of the shipment of samples, and distribution of resulting data were performed by EPA. The states of New York, South Carolina, Florida, and Texas chose to perform their own analyses and did not utilize the national contract. South Carolina and Florida provided their own QAPPs for review by the NCA QA staff and New York and Texas agreed to follow the requirements of the NCA QAPP. After review, the QAPPs submitted by South Carolina and Florida were accepted and each laboratory was conditionally approved to begin analyses. As a condition of each of these four states' cooperative agreements, each state laboratory will be audited during the time period 2003-2004.

## *National Contract Laboratories – NCA*

As part of the contract awards evaluation process each of the respondents were required to submit a QAPP for review as part of their proposal package. In their QAPP, they must either agree to adhere to the requirements of the NCA-QAPP or provide a plan with requirements that are equal to or greater than those described in the NCA-QAPP.

## Chemistry

The laboratory selected to perform the chemical analyses for the national contract agreed to adhere to the NCA-QAPP. The NCA national lab for 2000/2001 underwent a "Technical Systems Review" in January of 2001. The laboratory was commended for the efforts they were

expending to ensure the overall data quality. There were exemplary findings for sample tracking, QC Checklists, SOP's, Electronic data assembly, and laboratory personnel. Some concern was noted by the reviewers for validation of storage temperatures, documentation for comparisons of surrogate recoveries, and lack of access to raw inorganics data. Overall, the data received from this laboratory met or exceeded the requirements of NCA.

### **Toxicity**

The laboratory selected for the NCA national contract to perform the acute toxicity testing of sediments collected by NCA using *Ampelisca abdita* agreed to adhere to the requirements of the NCA-QAPP. A site visit of the national contract toxicity laboratory was conducted during December 2000. The facility and personnel were determined to be technically competent. The contractor had significant previous experience with the performance of the required toxicity tests through its contracting with the EMAP program in 1991-1994. The contractor underwent a "Data Quality Audit" during November 2001. The audit team was highly satisfied with the laboratory's overall technical capability to conduct the sediment toxicity tests on a high volume basis. The files were complete, orderly, and with minor exception, in compliance with the NCA-QAPP. The exceptions noted were: (1) some data entries made in pencil, and (2) the laboratory personnel were not initialing receipt of the samples on the log-in form.

### **Benthic Fauna**

The laboratory selected to perform the identification and enumeration of the benthic organisms collected by NCA agreed to adhere to the requirements of the NCA-QAPP. The laboratory's basic protocols met or exceeded those required by NCA; including, resorting of benthic samples, documentation on 10% of each technicians' samples (95% efficiency required), and taxonomic identifications being verified by a second taxonomist with an outside expert consulted for difficult identifications. The staff assigned to the project were determined to be technically competent and capable of performing the work.

### **Nutrients**

All water samples collected for determination of dissolved nutrients were analyzed at EPA/ORD/NHEERLs' Gulf Ecology Division. The analyses were performed with strict adherence to the NCA-QAPP. All analytical batches reported for inclusion in the NCA data base met or exceeded the requirements in the NCA-QAPP. A six point calibration curve with an  $r^2 \geq 0.95$ , internal check calibrant and external quality control samples were within the acceptable range of certification, and sample matrices were matched.

Five states in the northeast have chosen to perform their own nutrient analyses. In 2001, NCA Northeast Quality Assurance Coordinator established an inter-laboratory comparison study for nutrient analysis utilizing samples provided by NRC-Canada. Each laboratory was provided an unknown sample for analysis of inorganic nutrients. Laboratories are assessed by how close their results were to the NRC-Canada consensus values. For orthophosphate, 1 of the 5 laboratories agreed with the consensus values, 3 laboratories provided values close to the consensus value while one laboratory's results were not acceptable. For nitrite, 1 laboratory did

not submit a result, 3 laboratories provided values close to the consensus value while one laboratory's results were not acceptable. For nitrate/nitrite, 1 laboratory did not submit a result, 3 were in line and 1 was out. The laboratories were encouraged to continue participation in the NRC-Canada intercomparison for nutrients as part of their NCA QA programs.

## *Data Review*

All data received from the laboratories and field crews participating in the NCA program for 1999/2000 were reviewed prior to and during the data analysis phase. The NCA quality assurance team in the northeast developed a three-level QA review of data collected in their region (Appendix C). All of the data collected in the Northeast for 2000 was reviewed according to this procedure.

NCA west coast data collected in 1999 underwent an initial review for range checking, completeness, and consistency prior to placement into the database. Final review of the data was performed by the states and then discussed at a 2-day meeting between each state NCA participants and the NCA-West QA staff. The final version of the data set was then made available to the data analysts.

Southeastern and Gulf of Mexico data for 2000 was reviewed for range checking, completeness, and consistency by the NCA quality assurance staffs for these regions of the country. The datasets were checked for outliers and known relationships were tested. When these evaluations were completed, the data was supplied to the data analysts.

Analytical results from the national contract laboratories were reviewed as they were received. Each report was checked to ensure that the appropriate QC had been performed and that it met the requirements of the QAPP. When the data report was too voluminous to review by hand, the NCA data manager summarized the QA data and checked it in accordance the NCA-QAPP.